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DESIGN AND CONSTRUCTION OF A 1,4-DIOXANE VAPOR GENERATION SYSTEM

J.E. Reboulet and A.M. Lear

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Reboulet, James E.; and Lear, Amanda M.

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Naval Health Research Center Detachment
Environmental Health Effects Laboratory
2729 R St., Bldg 837
Wright-Patterson AFB, OH 45433

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ABSTRACT

A rodent, whole body inhalation study to characterize the biological effects of 1,4-Dioxane (dioxane) gas necessitated the development of a generation system capable of creating 6000 parts per million of dioxane in a 180 liter per minute air flow. Heat was required to counteract evaporative cooling of the dioxane while avoiding excessive heat so as not to increase temperatures in the exposure chamber. A variable alternating current transformer (Variac) was used to control two heating cords. The first warmed the glass generator body while the second heated the air passing through the generator. The temperatures used were sufficient to allow complete vaporization of the dioxane at 6000 ppm while not significantly increasing exposure chamber temperatures.

KEY WORDS AND PHRASES

1,4-Dioxane, Inhalation, Joules, BTU, Watts, Inhalation, Analyzer, Plenum, Vapor Exposure.

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INTRODUCTION

A generation system for 1,4-dioxane (dioxane) was designed to conduct a rodent, whole body vapor inhalation exposure. The study required a concentration of 6000 ppm of dioxane be maintained in an exposure chamber for a period of 6 hours. The exposure system operates at a flow rate of 180 liters per minute which requires evaporation of 3.76 ml of dioxane per minute to maintain a concentration of 6000 ppm as illustrated in equations 1 and 2.

(1) $$\frac{mg}{M^3} = \frac{6000\ L}{10^6\ L} * \frac{88.11*10^3 mg}{mole} * \frac{1\ Mole}{24.45\ L} * \frac{10^3 L}{M^3} = \frac{21600\ mg}{M^3}$$

(2) $$\frac{ml}{min} = \frac{21600mg}{M^3} * \frac{1}{10^3}\ \frac{g}{mg} * \frac{1}{10^3}\frac{M^3}{L} * \frac{180\ L}{min} * \frac{1}{1.034}\ \frac{ml}{g} = \frac{3.76\ ml}{min}$$

Dioxane is a non-volatile liquid due to its vapor pressure of 27 mm of mercury (Hg) at 20 °C (Specification sheet, Filo Chemical,New York,NY) and a heat of vaporization of 96.56 cal/gram at 744 mm of Hg (Vinson and Martin, 1963). Therefore, it requires the absorption of 375 calories per minute to counteract the cooling effects of evaporation (Equation3).

(3) $$Calories\ = \frac{96.56cal}{g} * \frac{1.034g}{ml} * \frac{3.76ml}{min} = \frac{375\ cal}{min}$$

The electrical energy required to supply 375 calories per minute is 26.12 watts (Equation 4).

(4) $$Watts\ = \frac{375\ calories}{minute} * \frac{4.18 joules}{calorie} * \frac{1\ watts{-}min}{60\ joules} = 26.12\ watts$$

The evaporation of the dioxane in the generator was achieved by passing 80 liters per minute (lpm) of warmed air through the generator. The generator output was then mixed with an additional 100 lpm of air flowing into the exposure chamber. The 26 watts of energy shown in equation 4 is equivalent to warming the air supplied to the generator 16 ˚ K from ambient (equation 5). The specific heat capacity of air at room temperature is 1.012 joules/gram-˚K, it density is 1.184 g/liter, and one joule equals 1 watt-sec.

(5) $$\wedge T = \frac{1\ min}{80\ liters} * \frac{1000\ \ liters}{1.184\ kg} * \frac{1\ kg{-}°\mathrm{K}}{1012\ watts{-}sec} * \frac{60\ sec}{1\ min} * 26.125 = 16.37\ °K$$

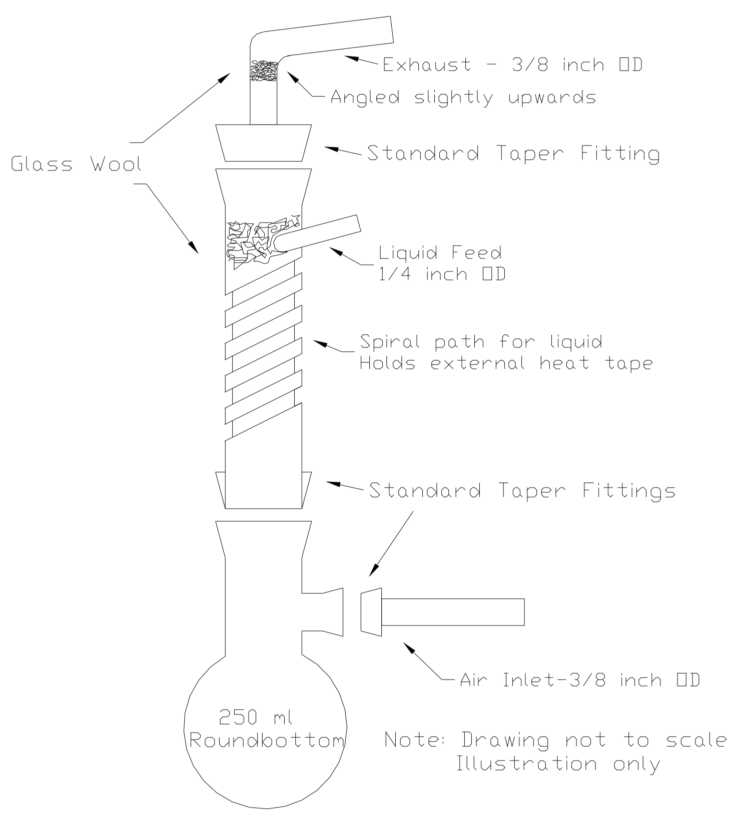
The kelvin (K) temperature scale is an extension of the degree Celsius scale down to absolute zero. Therefore, an increase of 16 ˚ K is the same as 16 ˚ C. With ambient temperature in the laboratory of approximately 20-23 °C a theoretical starting point for the generation system was to heat the air flowing through the generator to approximately 36-39°C.

Methods

System Design

A custom designed generation flask (Glassblowers.com INC., Turnersville, NJ) was constructed whose internal and external spiral column design increased residency time of the liquid dioxane in the heated zone and allowed convenient application of an external heating cord (Cole Parmer, #C-03122-60,Vernon Hills, IL). The cord profile is circular rather than flat which allowed it to fit the external spiral on the generator column. The air inlet and exhaust legs have a $^3/_8$ inch outside diameter (OD) so as allow them to be joined to standard $^3/_8$ inch Teflon tubing using Jaco compression fittings (Jaco Manufacturing, Berea, Ohio) and which allows easy interface to our existing exposure system. Standard taper joints were used on the air inlet and exhaust legs so that alternatively sized legs could be substituted in future experiments for connection to other exposure systems. The liquid feed tube was sized at $^1/_4$ inch OD so as to allow coupling through $^1/_4$ inch Jaco fittings. This size fitting easily accommodates a wide range of liquid flow rates. The gooseneck was angled upwards slightly to reflux any liquid which might reach that area. A round bottom flask was added to catch any excess input materials. Glass wool plugs were inserted to aid in liquid evaporation. The schematic representation of the generation system is shown in Figure 1.

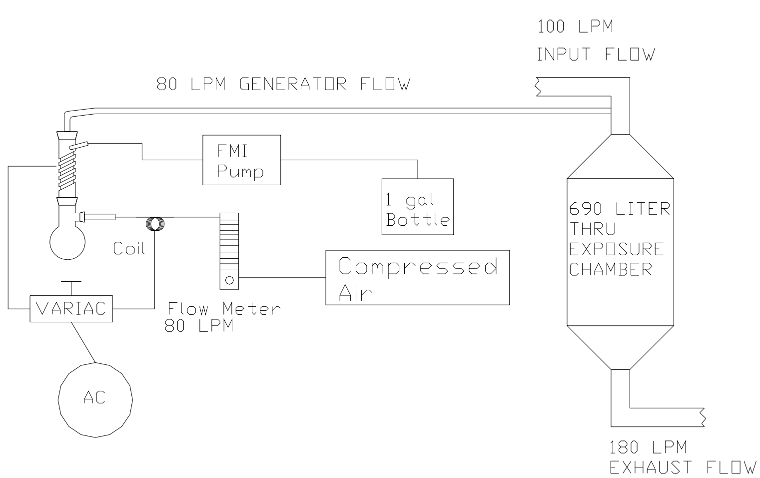
Figure 1



System Operation

Liquid was fed into the system via a FMI (FMI Inc, Syosett NY) model QG20 pump with a Q1CKC stainless steel pumping head and ceramic piston. The dioxane was drawn from a 4 liter glass jug via Teflon tubing and pumped at approximately 3.76 ml/minute into the top of the column. Heating cord, which has a circular profile, was wrapped in the spiral on the outside of the generator to warm the walls of the generator and assist evaporating the dioxane. The 80 lpm air flowing through the column was also heated. This heating was provided via a 5 foot long, $^{1}/_{2}$ inch soft copper tube (Plumbing Supply, Chico CA) bent into a 4 inch diameter coil, wrapped with heating tape (Cole Parmer, Vernon Hills IL) and inserted into the feed air line (Figure 2). Heating tape was used for this application as its flat profile made completely covering the surface of the coil easier. A variable Alternating current transformer (ISE Inc., VARIAC, Cleveland OH) was connected to both the heat tape and heat cord by use of a 3 way adaptor. The voltage output of the VARIAC was adjusted to heat the coil to approximately 40-44 ˚C as measured externally with a thermocouple thermometer equipped with a type K thermocouple (Omega, model 600, Stamford CT). At this setting and with an input air flow rate of 80 liters per minute (lpm) controlled with a Dwyer flow meter (Dwyer, RMB-54D-SSV,Michigan City, IN) the generator delivers air at a constant temperature of 36 ˚C as measured at the gooseneck. This is the temperature necessary as predicted by theoretical calculations (equation 5) to yield complete evaporation of the dioxane. To help maximize the surface area of the dioxane, and to aid rapid evaporation in the generator, a glass wool plug approximately 2 inches long was inserted in the area where the dioxane enters the column. The glass wool spreads the dioxane out across the diameter of the column allowing maximum interaction and absorption of heat from the warmed air passing through it. To minimize development of aerosol droplets due to air blowing past a liquid interface, another glass wool plug of 1-2 inches in length was placed in the gooseneck to capture these droplets and permit final evaporation.

Figure 2



RESULTS

Using a temperature of approximately 44 °C for the copper coil, as well as heating the generator body at the same VARIAC setting, gave a generator air flow temperature of approximately 36 °C. at the outlet of the gooseneck fitting. At this temperature and a flow rate of 80 lpm concentrations of 6000 ppm of dioxane could be maintained in the exposure chamber for periods in excess of 6 hours with no observable liquid getting into the round bottom collection flask. Under these conditions the exposure chamber only increased in temperature by approximately 2 °C above ambient.

DISCUSSION

During the initial testing phase evaporation of 4 ml / minute of dioxane was attempted by using just a heating cord around the glass generator column. It quickly became apparent that the glass of the column was not transmitting the heat rapidly enough to evaporate all the dioxane as the surplus would run down the inside channel and drop into the 250 ml catch flask. To supply additional heat to the input/generator air a 4 inch diameter copper coil was formed from a five foot length of ½ inch soft copper tubing. This coil was wrapped with heating tape and put into the system. This improved the overall evaporation rate but not enough to obtain complete vaporization at a rate of 4 ml/min. The problem was that too much of the heated air was simply lost flowing past the dioxane droplets moving down the inner wall of the generator. The surface area presented by the dioxane needed to be increased. This was accomplished by adding a plug of glass wool which dispersed the droplet over the entire cross sectional area of the generator column. The addition of the glass wool allowed complete evaporation at a rate of 4 ml / minute. The final step was to lower the temperature across the copper coil and generator body to yield 36°C air temperature at the exit of the gooseneck. The lowest usable temperature was desired to keep the increase in temperature in the exposure chamber at a minimum. At this temperature, an evaporation rate of 4 ml / minute was maintainable. Since the desired evaporation rate for 6000 ppm was 3.76 ml / minute this gave some flexibility in actual operation of the system.

CONCLUSION

The glass generation system, coupled with heated air, and glass wool dispersal of the liquid, allowed the generation of 6000 ppm of dioxane for 6 hours. This system also allows for generation of lower concentrations of dioxane and could serve as a general purpose generator for other volatile liquids.

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